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Indian Standard

SPECIFICATION FOR HEXACHLOROETHANE

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110001

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SPECIFICATION FOR HEXACHLOROETHANE

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Indian Standard

SPECIFICATION FOR HEXACHLOROETHANE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 30 December 1974, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Chemical Division Council.

0.2 Hexachloroethane is used in the metallurgical industry for casting of aluminium and degassing of magnesium and other non-ferrous metals. It is also used in the manufacture of smoke screens and grenades for defence purposes. It also finds use as a plasticizer for cellulose esters in place of camphor; in the formulation of high pressure lubricants; as an anthelmintic in veterinary medicine; and in the formulation of various fungicides, insecticides and monthrepellants.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for hexachloroethane.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of colourless to pale brown crystalline solid free from visible impurities. It shall be non-flammable and practically insoluble in water.

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. Reference to the relevant clauses of the appendix is given in col 4 of the table.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR HEXACHLOROETHANE

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	Melting point	183 to 187°C	A-2
ii)	Ash and gritty matter, percent by mass, *Max*	0·03 (of which not more than 0·01 percent shall be retained on 63 micron IS Sieve; grit shall completely pass 250 micron IS Sieve)	A-3
iii)	Matter insoluble in ethanol, percent by mass, *Max*	0·05	A-4
iv)	Moisture, percent by mass, *Max*	0·05	A-5
v)	Alkalinity (as Na_2CO_3), percent by mass, *Max*	0·01	A-6
vi)	Free chlorine	To pass the test	A-7
vii)	Water soluble chlorides (as NaCl), percent by mass, *Max*	0·02	A-8

3. PRECAUTIONS IN HANDLING AND STORING

3.1 Hexachloroethane sublimes readily without leaving any residue and, therefore, the containers should be kept air-tight.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed, stored and transported in mild steel drums with suitable liners or polythene bags.

4.2 The material shall be supplied in accordance with the marking and delivery instructions given by the purchaser.

4.3 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name and mass of the material in the container;

b) Name of the manufacturer and recognized trade-mark, if any; and

c) Lot or batch number in code or otherwise.

4.3.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 Representative samples of the material shall be drawn and their conformity to the standard judged as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2 and *Table* 1)

METHODS OF TEST FOR HEXACHLOROETHANE

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF MELTING POINT

A-2.1 Procedure — Determine the melting point according to the method prescribed in IS : 5762-1970† using the thermometer prescribed under **A-2.1.1**.

A-2.1.1 *Thermometer* — of the mercury-in-glass type with a range from 85 to 215°C, graduated at every 0·2 deg, with partial immersion mark at 100 mm, and having a maximum error of ± 0·2 deg. A thermometer of other suitable range and accuracy may also be used.

A-3. DETERMINATION OF ASH AND GRITTY MATTER

A-3.0 Outline of the Method — A known mass of the sample is ignited at dull red heat and the percentage ash is known. Any grit is observed

*Specification for water, distilled quality (*revised*).

†Methods for determination of melting point and melting range.

by means of exerting pressure on glass slide. About 100 g of the sample is ignited and digested with aqua regia. The grit is poured in water, washed, dried, weighed and classified.

A-3.1 Apparatus

A-3.1.1 *Silica Dish*

A-3.1.2 *Test Sieves* — 250-micron and 63-micron (*see* IS : 460-1962*).

A-3.1.3 *Microscope Slides* — two.

A-3.1.4 *Camel Hair Brush*

A-3.1.5 *Hand Glass* — magnification × 6.

A-3.2 Reagents

A-3.2.1 *Aqua Regia* — Mix 25 ml of concentrated nitric acid (relative density 1·42) with 75 ml of concentrated hydrochloric acid (relative density 1·18).

A-3.3 Procedure — Ignite the silica dish at dull red heat. Allow it to cool in a desiccator and weigh. Weigh 20 g of the sample in the dish and gently burn it, a little at a time. Ignite the residue at dull red heat, allow to cool in a desiccator and reweigh. Place the residue between the microscope slides and exert pressure on the latter by finger and thumb. Note from the feel and sound if any grit is present. If the presence of grit is detected, determine the amount by the following procedure:

Weigh 100 g of the sample in the silica dish and gently burn it, a little at a time and finally ignite at dull red heat. Allow the ash to cool and add 10 ml of aqua regia. Digest the ash for 10 minutes, pour the contents of the dish into 150 ml of water contained in a 250-ml beaker and wash any residue from the dish into the beaker, using a jet of water. Decant most of the water from the beaker and then wash the grit on to 63 micron IS Sieve with the aid of the brush, if necessary. Dry sieve the grit in an oven, then gently brush on the sieve so that any fine particles pass through and then transfer that retained on the sieve to a tared weighing bottle. Dry the bottle at 100 ± 2°C and reweigh to the nearest 0·5 mg. Transfer the grit to 250 micron IS Sieve and brush gently. Examine the sieve under a hand glass (× 6) to see if any grit is retained.

*Specification for test sieves (*revised*).

A-3.4 Calculation

A-3.4.1 Ash, percent by mass $= \frac{M_1}{M_2} \times 100$

where

M_1 = mass in g of the residue after ignition, and

M_2 = mass in g of the sample taken for the test.

A-3.4.2 Gritty matter retained on 63-micron IS Sieve, percent by mass $= (M_3 - M_4)$

where

M_3 = mass in g of the weighing bottle and grit remaining from 100 g of the sample, and

M_4 = mass in g of the weighing bottle.

A-3.4.3 Report whether the grit completely passes through 250-micron IS Sieve or not.

A-4. DETERMINATION OF MATTER INSOLUBLE IN ETHANOL

A-4.1 Reagent

A-4.1.1 *Ethanol* — 95 percent.

A-4.2 Apparatus

A-4.2.1 *Sintered Glass Crucible* — Grade No. 4.

A-4.2.2 *Vacuum Device for Filtration*

A-4.3 Procedure — Dissolve 15 g of the sample in 100 ml of ethanol by warming. Filter through a sintered glass crucible attached with vacuum device. Wash with warm ethanol, dry in an oven at 100 ± 2°C and weigh.

A-4.4 Calculation

Matter insoluble in ethanol, percent by mass $= \frac{M_1}{M_2} \times 100$

where

M_1 = mass is g of the residue, and

M_2 = mass in g of the material taken for the test.

A-5. DETERMINATION OF MOISTURE CONTENT

A-5.0 General — Moisture is determined by the Karl Fischer method.

A-5.1 Procedure — Take 10 g of the material and dissolve in 25 ml of carbon tetrachloride and methanol mixture (1 : 1). Determine the moisture content by the procedure given in IS : 2362-1973*.

A-6. ACIDITY OR ALKALINITY

A-6.0 Outline of the Method — A known mass of the material is dissolved in carbon tetrachloride (or any other suitable solvent), shaken with neutralized distilled water, then treated with excess standard hydrochloric acid using bromophenol blue solution as indicator and back titrated with standard sodium hydroxide solution. The end point is noted when the colour of the aqueous layer matches that of the neutralized water. From the difference of standard hydrochloric acid and standard sodium hydroxide used, the alkalinity is expressed as percentage sodium carbonate. A blank determination is also done.

A-6.1 Apparatus

A-6.1.1 *Glass Stoppered Flasks* — 250-ml capacity, two.

A-6.2 Reagents

A-6.2.1 *Standard Sodium Hydroxide Solution* — 0·1 N.

A-6.2.2 *Rectified Spirit — See* IS : 323-1959†.

A-6.2.3 *Standard Hydrochloric Acid* — 0·1 N.

A-6.2.4 *Bromophenol Blue Indicator* — Dissolve 0·2 g of bromophenol blue in 3 ml of 0·1 N sodium hydroxide solution, and dilute to 100 ml with rectified spirit.

A-6.2.5 *Neutralized Distilled Water* — Measure 100 ml of distilled water into one of the flasks. Add 1 ml of bromophenol blue indicator and, if necessary, neutralize by dropwise addition of the sodium hydroxide solution or standard hydrochloric acid until a neutral (green) tint is obtained.

A-6.3 Procedure — Place 50 ml of carbon tetrachloride in a 250-ml glass stoppered conical flask. Add 20 g of sample and shake to dissolve. Add 100 ml of neutralized water and shake well and note any change in colour towards yellow, indicating the presence of acidity. If not acidic add in excess standard hydrochloric acid, shake vigorously and allow the liquid layers to separate. Titrate the excess of hydrochloric acid with standard sodium hydroxide solution, gently swirling the flask and adding a few drops at a time. Note the end point when the colour of the aqueous layer, after allowing the layers to separate, matches that of the neutralized water.

*Determination of water by the Karl Fischer method (*first revision*).
†Specification for rectified spirit (*revised*).

A-6.4 Calculation

Alkalinity (as Na_2CO_3), percent by mass = $0{\cdot}0265 \times (V_1 - V_2)$

where

V_1 = volume in ml of standard hydrochloric acid, and

V_2 = volume in ml of standard sodium hydroxide solution required for the back titration.

A-7. TEST FOR FREE CHLORINE

A-7.0 Outline of the Method — The material is shaken with 3, 3'-dimethylnaphthidine solution and the colour developed, if any, is noted.

A-7.1 Reagents

A-7.1.1 *Glacial Acetic Acid*

A-7.1.2 *3, 3'-Dimethylnaphthidine Solution* — Dissolve 0·01 g of finely ground 3, 3'-dimethylnaphthidine in 5 ml of glacial acetic acid and dilute rapidly to 200 ml. Store the solution in the dark.

A-7.2 Procedure — To 10·00 g of the material contained in a measuring cylinder, add 20 ml of water and 5 ml of the 3, 3'-dimethylnaphthidine solution. Shake the cylinder for 30 seconds.

A-7.2.1 The material shall be regarded to have passed the test if no pink colour is developed.

A-8. DETERMINATION OF WATER SOLUBLE CHLORIDES

A-8.1 Reagents

A-8.1.1 *Concentrated Nitric Acid* — conforming to IS:264-1968*.

A-8.1.2 *Standard Silver Nitrate Solution* — 0·1 N.

A-8.1.3 *Nitrobenzene*

A-8.1.4 *Standard Ammonium Thiocyanate Solution* — 0·1 N.

A-8.1.5 *Ferric Ammonium Sulphate Indicator* — approximately 5 percent.

A-8.2 Procedure — Weigh accurately about 20 g of the material, dissolve in water and neutralize with concentrated nitric acid and then add about 5 ml in excess. Boil the solution to expel any dissolved carbon dioxide gas, cool and add 10 ml of standard silver nitrate solution.

*Specification for nitric acid (*first revision*).

Add 3 ml of nitrobenzene, shake vigorously and titrate with standard ammonium thiocyanate solution using ferric ammonium sulphate indicator.

A-8.3 Calculation

$$\text{Water soluble chlorides (as NaCl), percent by mass} = \frac{5{\cdot}846\ (10\ N_1 - V N_2)}{M}$$

where

N_1 = normality of standard silver nitrate solution,

V = volume in ml of standard ammonium thiocyanate solution consumed in the titration,

N_2 = normality of standard ammonium thiocyanate solution, and

M = mass in g of the material taken for the test.

APPENDIX B

(*Clause* 5.1)

SAMPLING OF HEXACHLOROETHANE

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken at a place protected from damp air, dust and soot.

B-1.2 Sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling, shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost but not completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All containers in a single consignment belonging to the same batch of manufacture shall be grouped together to constitute a lot.

B-2.2 For ascertaining the conformity of the lot to the requirements of the specification, samples shall be tested for each lot separately. The number of samples to be selected shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

Lot Size	No. of Containers to be Selected
N	n
(1)	(2)
Up to 50	3
51 ,, 100	4
101 ,, 150	5
151 ,, 300	6
301 ,, 500	7
501 ,, 800	8
801 ,, 1 000	9
1 001 and above	10

B-2.3 These containers shall be selected at random. In order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-3. TEST SAMPLE AND REFEREE SAMPLE

B-3.1 From each of the containers selected as in **B-2.2**, draw with an appropriate sampling instrument small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

B-3.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

*Methods for random sampling.

B-3.3 From samples (*see* **B-3.2**) representing different containers selected in **B-2.2**, small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.4 The remaining portion of the material in the sample (*see* **B-3.2**) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

B-3.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **B-1.7.**

B-3.6 The referee samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-4. NUMBER OF TESTS

B-4.1 Melting point, matter insoluble in ethanol and alkalinity shall be tested on each of the individual samples.

B-4.2 Tests for all other characteristics given in Table 1 shall be conducted on the composite sample.

B-5. CRITERIA FOR CONFORMITY

B-5.1 Individual Samples — The lot shall be declared as conforming to the requirements tested on individual samples if all the test results satisfy the corresponding requirements given in Table 1.

B-5.2 The lot shall be declared as satisfying the remaining requirements if all the test results on the composite sample satisfy the corresponding requirements given in Table 1.

B-5.3 For declaring the conformity of the lot to the requirements of this specification **B-5.1** and **B-5.2** shall be satisfied.

INDIAN STANDARDS
ON
ORGANIC CHEMICALS (MISCELLANEOUS) MATERIALS

IS:

245-1970 Trichloroethylene, technical (*second revision*)
501-1963 Oxalic acid, technical and analytical reagent (*revised*)
716-1970 Pentachlorophenol (*first revision*)
717-1969 Carbon disulphide, technical (*first revision*)
718-1970 Carbon tetrachloride (*first revision*)
869-1969 Ethylene dichloride (*first revision*)
880-1956 Tartaric acid
3321-1965 Formaldehyde solution
4105-1967 Styrene (vinyl benzene)
4306-1970 Hexamethylenetetramine (hexamine) (*first revision*)
4566-1968 Methylene chloride (dichloromethane), technical
5149-1969 Maleic anhydride, technical
5158-1969 Phthalic anhydride, technical
5254-1969 Acetanilide
5271-1969 Paraformaldehyde
5295-1969 Ethylene glycol
5295-1969 Chloroform, technical and analytical
5297-1969 Perchloroethylene (tetrachloroethylene), technical
5341-1969 Benzyl chloride, technical
5464-1970 Citric acid, monohydrate
5573-1969 Ethylene oxide
5591-1969 Chlorobenzene
5592-1969 Monochloroacetic acid
5992-1969 *p*-Dichlorobenzene, technical
6393-1971 α phenylacetamide
6412-1971 Benzoyl chloride, technical
6515-1972 Sodium pentachlorophenate, technical
6712-1972 *o*-Dichlorobenzene
6716-1972 Benzoic acid, technical
6718-1972 Phenoxyacetic acid
6768-1973 *m*-Aminophenol
6775-1973 Ethyl chloride, technical
6971-1973 2-Ethyl hexan-1-ol
6972-1973 Benzo-trichloride
7134-1973 Diphenyl
7135-1973 Dimethyl sulphate, technical

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